# HIGH-PRESSURE RINSE AND CHEMICAL POLISH OF A SPOKE CAVITY

M.P. Kelly, K.W. Shepard, M. Kedzie, Argonne National Laboratory,
Argonne, IL 60439, USA

*Abstract*

We present here a description of new automated high-pressure rinse and chemical polishing facilities used for surface processing of superconducting niobium drift-tube cavities at Argonne National Laboratory (ANL). The expanded infrastructure is necessary to develop and process new types of cavities for the RIA (Rare Isotope Accelerator) project and has also been used in surface reprocessing of split-ring structures in the existing ATLAS accelerator. Results of surface reprocessing on a 350 MHz niobium spoke cavity followed by high power rf-conditioning with a recently acquired 5 kW transmitter have produced accelerating gradients of 7 MV/m at T = 4.3 K and 10 W of input rf power.

## 1 INTRODUCTION

Prototype cavities for a superconducting (SC) multi-ion driver linac [1] for the Rare Isotope Accelerator (RIA) [2] are being developed at ANL to span the velocity region, $0.2<\beta<0.6$ where until recently little work had been done [3]. A pair of prototype spoke cavities with $\beta$=0.3 and $\beta$=0.4 have been built, processed and recently tested successfully [4,5]. A prototype $\beta$=0.4 double-cell spoke cavity is currently under construction to be followed shortly by a $\beta$=0.15 two-gap quarter-wave cavity [6]. A half-wave resonator is planned with peak beta intermediate to the quarter-wave and spoke cavities. This paper reports on new facilities at ANL built to accommodate evolving cavity designs. An additional benefit is the ability to high-pressure rinse (HPR) existing ATLAS resonators. The first test showed dramatically improved accelerating gradients following a high-pressure rinse on an ATLAS split-ring cavity and a full recovery of the original performance after 17 years of operations. High-pressure rinse appears to provide the same benefits for drift-tube structures that have been observed with elliptical cell cavities.

## 2 HIGH-PRESSURE RINSE

A new automated high-pressure rinse (HPR) system consisting of a high-pressure pump, a spray wand, and custom spray nozzles has been installed at ANL (see Figure 1). The system has been used as the final step in the surface preparation of SC niobium cavities. The principal specifications of the system are listed below.

### *2.1 Specifications*

- System Cost – $12,000 US
- Pump Unit – Karcher HD1090
- Output Pressure – 0 to 3000 PSI
- Flow Rate – 0 to 20 liters/minute
- Water Supply – 500 gallon storage tank ultra-pure deionized water
- Wetted parts – 316 and 304 stainless steel, nylon, polyethylene and viton.
- Filtration – 0.5 mm sintered stainless steel from Swagelock and 0.04 mm PROPOR PES from Domnick-Hunter.
- Environment – Monitored class-100 clean area.
- Wand Motion – Continuous rotation and translation up to 91 cm. on carriage from StoneAge Waterjet Tools.
- Control – Fully programmable wand speed, direction and distance using a PC running LabView.

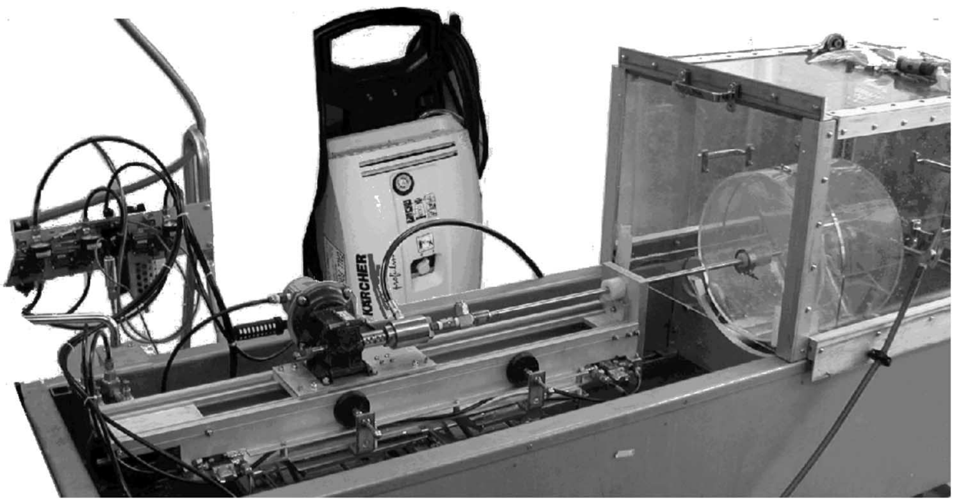


Figure 1: A new automated high-pressure rinse apparatus for the surface preparation of SC niobium cavities at ANL.

### *2.2 Discussion of the Apparatus*

Components of the high-pressure rinse system are mostly 'off the shelf'. Exceptions are the support carriage for the spray wand, the wand and spray nozzles constructed by StoneAge Waterjet Tools and a stainless steel filter housing suitable for pressures up to 300 bar purchased from Domnick-Hunter. The 0.5 μm and 0.04 μm filters on the high pressure side of the pump were added to remove any (brass) contamination introduced by the pump itself. Brass fittings on the pump and the supplied rubber hose have been replaced by 316 stainless steel fittings and a high-pressure nylon bore hose all from Swagelock.

The horizontal orientation of the wand permits good drainage from the spoke and split-ring cavity coupling ports. Water flow rates are read with a magnetic induction paddle wheel and the pressure is read using a 0-3000 PSI pressure transducer both from Omega.com.

### *2.3 High-pressure Rinse Tests*

Initial tests of the high-pressure rinse system were performed on small niobium samples, some electro-polished and others chemically etched. Samples were pre-cleaned with ethanol and acetone. A single jet with a pressure of 2100 PSI was directed toward the samples for 5 minutes at a distance of 2.5 cm. Analysis of samples under an optical microscope showing surface detail down to 1 $\mu m^2$ revealed a large (>99%) reduction in surface particulates and no apparent surface damage from the high-pressure water.

In order to help rule out contamination due to rinse water the analytical chemistry group at ANL performed detailed chemical analyses of the deionized water flowing into and out of the HPR system. Results showed no dissolved solids in the water to 1 part in $3x10^6$ by weight and no detectable dissolved anions for 28 different species at the level of 10-50 ppm depending on the ion. The water resistivity both into and out of the system was measured to be ~18 MΩ cm.

Niobium cavities were subsequently rinsed at a nozzle pressure of 1700 PSI. In one case an extremely low residual surface resistance was achieved following a 1-hour rinse (2.7 nΩ for a 97 MHz split-ring) providing a strong validation of the procedure. Water pressures used here are somewhat higher than those reported elsewhere, however, it is important to note that the relevant physical quantity, the jet velocity, is strongly affected by the nozzle size and geometry for turbulent flow. The jet velocity here was measured to be 150 m/s for a nozzle pressure of 1700 PSI.

## 3 BUFFERED CHEMICAL POLISH

### *3.1 Description of Apparatus*

A new setup for performing buffered chemical polish (BCP) has been installed at ANL to chemically etch the interior of cavities with closed geometries where electropolishing is not practical. The facility consists of gravity fill tanks for acid and water, a water cooling tank (not shown in Figure 2) to moderate the cavity temperature during the etch, and a recirculation pump to agitate the acid solution.

Tanks for acid and water are standard PVDF construction. For plumbing both PVDF and Teflon are used. Pneumatically controlled Teflon diaphram pumps and PVDF valves are used to control acid and water transport. The entire apparatus rests inside a chemistry hood and is operated using a remote control box just outside the hood.

BCP is performed using a standard 1:1:2 solution of 48% hydrofluoric, 69% nitric and 85% phosphoric acids obtained premixed in 55 gallon containers. The acid temperature is maintained in the range $12^O$ - $18^OC$ by an external water-cooling bath and acid is recirculated through the beam ports at a rate of 20 liters/minute to help remove the evolved gas.

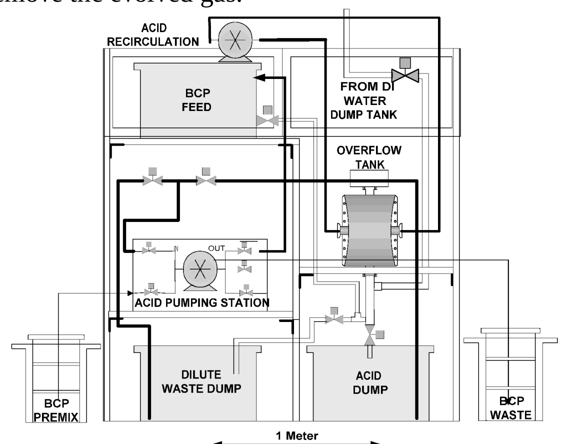


Figure 2: An apparatus for chemical polish at ANL.

### *3.2 Anomalous BCP Etch Rates*

Initial tests were performed with small volumes of standard 1:1:2 BCP mixed by hand from bottles of fresh reagent. For solutions ~10 minutes old extremely high etch rates were observed. In one test a small niobium sample with dimensions of 2.5x2.5x0.32 cm. was placed in 150 ml of fresh BCP in a small plastic beaker all initially at room temperature. The niobium surface area-to-acid ratio is roughly the same as that for the spoke cavity during BCP. The mixture exhibited a potentially dangerous increase in temperature of 22 $^oC$ to 52 $^oC$ in 1 minute. Some of the unused BCP solution was stored overnight at 5 $^oC$ and tested again the following day, this time exhibiting only a small temperature rise and an etch rate of ~1 micron/minute at 15 $^oC$, similar to the premixed solution in the 200 liter barrels. This equilibration time may normally go unnoticed since the quantities of acid needed to polish a cavity, and therefore, the time between mixing and use tend to be greater than in these tests. However, users should be aware that the BCP solution requires time to equilibrate.

## 4 CAVITY RESULTS

### *4.1 Prototype Spoke Cavity*

Initial processing of the spoke cavity (see Figure 3) after construction included a heavy ~150 micron electropolish just prior to the final closure weld followed by a very light chemical polish. The cavity was rinsed by filling with clean deionized water.

Tests following the initial cooldown to 4.5 K were performed with 110 W of rf power for pulse conditioning. The observed low level Q was $6x10^8$ corresponding to a surface resistance of 137 n$\Omega$ with a maximum accelerating gradient just over 4 MV/m as shown in Figure 4. Additional rf pulse conditioning with a new 5 kW transmitter lead to accelerating gradients up to 6.4 MV/m (green points).

In an effort to further increase performance the spoke cavity was treated in new chemical polish and high-pressure rinse facilities at ANL. A total of 123 μm of niobium was removed from the inside surface of the cavity during two separate BCP treatments of 1 hour each. This was followed by a 1 hour high-pressure rinse and 48 hours of drying under a nitrogen purge in a class-100 clean area.

Figure 3: A 350 MHz β=0.4 prototype spoke cavity

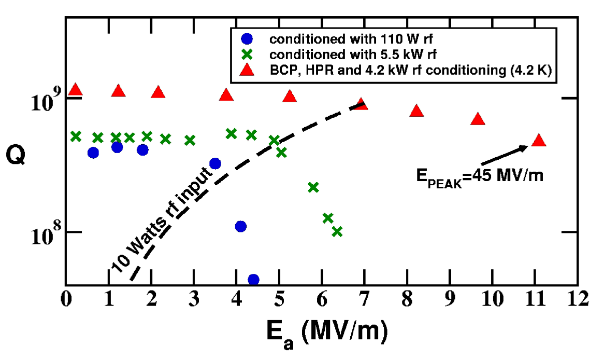


Figure 4: Results for the spoke cavity at 4.2 K following a high-pressure rinse, buffered chemical polish and rf conditioning (top) compared to earlier results with only rf conditioning (bottom).

Cavity results at 4.2 K are shown in Figure 4. The Q at 4.2 K for low input power is better than $10^9$ and field emission has been dramatically reduced even for the highest stable accelerating field of 11.5 MV/m ($E_{PEAK}$=46 MV/m). The design goal of $E_{ACC}$=5 MV/m is achieved with 5 W of input rf power at 4.2 K. An effort to rinse cavities in the existing ATLAS is underway. First results show dramatic improvements as seen in Figure 6.

Figure 5: An ATLAS 97 MHz split-ring cavity.

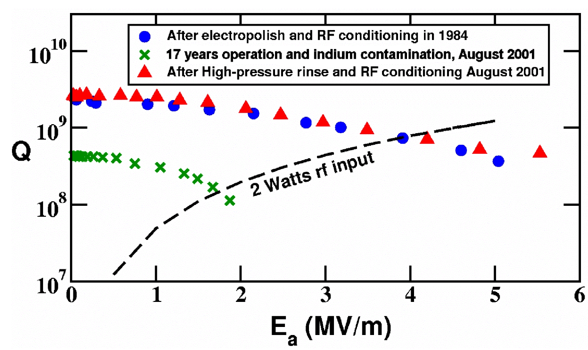


Figure 6: A 17-year-old split-ring cavity tested after recent high-pressure rinsing and rf pulse conditioning exhibits the highest Q (>$6x10^9$ at 2 K), highest cw accelerating fields (6.8 MV/m at 2 K) and lowest surface resistance ($R_{RES}$=2.7 n$\Omega$) observed in this class of resonator. Initially one of the best performers 1984, this cavity had indium debris introduced into the interior which degraded performance (crosses). Performance fully recovered following the rinse.

## 5 ACKNOWLEDGEMENTS

The authors acknowledge Gary Zinkann and Javier Figueroa for their help with cavity cold tests. This work was supported by the U.S. Department of Energy, Nuclear Physics Division, under contract number W-31-109-ENG-38.

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